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STUDY OF
LIQUID OXYGEN CONTAMINATION

R. H. Foster and S. T. Stoy

AIR PRODUCTS and CHEMICALS, INC.

Allentown, Pennsylvania

A. P. C. I. Research Project No. 03-9-2881

Contract No. AF 33(616)-6730

JANUARY 1961

Directorate of Rocket Propulsion

AIR FORCE FLIGHT TEST CENTER

AIR RESEARCH AND DEVELOPMENT COMMAND

UNITED STATES AIR FORCE

Edwards Air Force Base, California

Copy No. 28

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Progress Report No. 6

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ABSTRACT

This progress report describes the work performed on filtration of solid carbon dioxide particles from liquid oxygen and on ignition of hydrocarbon films in oxygen atmospheres. During this period an investigation of commercial filter assemblies satisfactory for liquid oxygen service was undertaken.

The results of this work indicate that a maximum safe level of hydrocarbon (hexadecane) film contamination is 100 milligrams per square foot.

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I. INTRODUCTION

In July, 1959, Air Products and Chemicals, Inc. was awarded the subject contract for the examination of possible liquid oxygen contamination problems. This work was fully reported (1, 2, 3, 4, 5). In May, 1960 Supplemental Agreement No. 1 was awarded, extending work under this contract in certain phases. These phases were as follows:

"Phase IV - Sources and Mechanisms for Ignition

"The Contractor shall conduct an experimental program to study the phenomenon of static electricity in liquid oxygen systems. This shall include investigation of such variables as rates of charge build-up, system geometry, and dynamics and the nature and amount of impurities which are likely to be present. Other possible sources and mechanisms for ignition which might exist in oxygen systems will also be studied. Primary effort shall be placed on the analytical aspect.

"Phase V - Factors Related to Solid Contaminants

"The Contractor shall determine the effects of solid matter in liquid oxygen as it pertains to the use in missile systems. This phase shall include the following areas:

1. Determination of the factors relating to the agglomeration of small crystals or particles of insoluble contaminants such as carbon dioxide and the heavier hydrocarbons.

2. Determination of the effect of solid particles (rust, sand, lint, etc.) on oxygen systems, and recommend limits for various operating conditions. Particle size and quantity are to be considered.

3. Recommendations of proper operational conditions to reduce or eliminate all solid material shall be made.

4. Using the data obtained from paragraphs 1 through 3 above, recommend specification limits for the various contaminants, and suitable quality assurance provisions (See Phase III).

5. Conduction of a thorough investigation to determine the relative merits of the different types of filters for removing all types of solid material (crystal, agglomerated particles, granular and fibrous matter) from liquid oxygen. Specific filters shall be recommended. If commercially available equipment is not suitable, design criteria shall be developed.

"Phase VI - Cleaning and Purification

"During the various handling and transfer operations, clean liquid oxygen can become contaminated because of dirty equipment. To assure proper maintenance of oxygen quality, the Contractor shall:

1. Develop uniform cleanliness standards for oxygen equipment. (Development of cleaning agents, equipment and techniques are beyond the scope of this contract.)

2. Develop and recommend suitable means of inspection and surveillance of oxygen systems to assure proper cleanliness is maintained.

3. Study methods of removing contamination from liquid oxygen at the storage site, and make recommendations to the use of such techniques to reduce the disposal of contaminated oxygen, and to reduce the need for stringent cleaning requirements of oxygen systems.

"Phase VII - Safety Standards for Use of High Pressure Gases

"The Contractor shall prepare a document covering the safe handling, storage, shipping, and use of high pressure gases. The format shall conform to that of the "Liquid Propellant Safety Manual", published by the Liquid Propellant Information Agency for the Office of the Assistant Secretary of Defense (R&E). The gases to be considered are:

1. Oxygen
2. Nitrogen
3. Helium
4. Other gases as mutually agreed upon by AFFTC and Air Products, Incorporated."

Progress Report Number 5 of October, 1960 presented the opening phases of programs of carbon dioxide filtration from liquid oxygen and of hydrocarbon film ignition (6). The results of this work showed that a correlation apparently existed between filtration efficiency and filtration rate. The information obtained on film contamination indicated a higher possible limit of film-type hydrocarbon contamination than previously allowed (100 milligrams per square foot) (7).

This report, No. 6, continues both phases of that work. It also presents the recommendations for the use of the commercial filters in liquid oxygen service for the removal of all types of solid materials. During this period, some of the personnel who were involved in work under this contract were temporarily assigned to complete arrangements for the analytical phase of the program to be performed for a three-month period at Cape Canaveral Missile Test Center, Cape Canaveral, Florida.

II. DISCUSSION

A. Solid Carbon Dioxide Filtration

1. Experimental Apparatus

The experimental apparatus used to obtain the information presented in this report was essentially that shown in Figures 1 through 8 of Progress Report No. 5 (6). The Reynolds number and carbon dioxide concentration had been observed for 43 runs with 5-micron and 10-micron filter types. The procedure and the results of these 43 carbon dioxide filtration tests with several different types of filters were summarized in Report No. 5, October, 1960 (6). The correlation of carbon dioxide concentration in the filtrate versus Reynolds number at the filter surface was postulated. From October, 1960 through January, 1961, the work on 17 additional tests was completed. These results agree with the correlation under development in the previous report (6). They are reported in Table I and are shown in Figure 1. In addition to tests 44 through 60, we are presenting additional data developed by Philips and Snyder of Air Products and Chemicals, Inc. at the Denver facility in February and April, 1960 (8). These tests were run on the standard 75 T/D liquid oxygen generating unit, a type which is used at missile installations, particularly at Cape Canaveral and Santa Susana. As shown in Figure 1, the data from the large-scale work carried out at Denver agree with the laboratory-scale work conducted at

Air Products and Chemicals Research and Development Department at Emmaus, Pennsylvania. Figure 1 shows the data on concentration of carbon dioxide after filtration plotted against Reynolds number in the filter assembly. The Reynolds number is a flow correlation of $Re = 4w D_h/A\mu$, where: w = flow rate, lb/hr; D_h = hydraulic diameter, feet; A = crossectional area, sq ft; and μ = viscosity, lb/ft sec.

Tests 44 through 60 were conducted to investigate filtration of a boiling liquid and of a turbulent liquid. In the boiling tests, a portion of the liquid feed to the filter assembly was vaporized and warmed by heat exchange with nitrogen at atmospheric temperature. It was then recombined with the liquid stream. Because of the difficulties of controlling liquid oxygen and gaseous oxygen flows at low rates in small equipment, this procedure actually developed a slugging type of flow wherein for a time the flow was 100 percent liquid. After a period of time the flow became 100 percent gas. This cycling continued throughout the tests.

All previously reported runs were made with a porous stainless steel filter element of nominal 7-micron pore size. Runs 44 through 48 were done with sub-cooled liquid oxygen. Runs 44 and 45 were made without vibration and runs 46, 47, and 48 were done with external vibration of the filter assembly.

Runs 49 through 60 were made with turbulent liquid in two-phase flow. A side take-off was added to the filter assembly to increase the flow turbulence in the filter assembly. The main portion of the flow passed out the side take-off without going through the filter element. Thus, only a small portion of the total flow passed through the filter. Since the unfiltered liquid oxygen from the side take-off was dumped into the liquid nitrogen bath, the nitrogen bath level varied throughout each run.

2. Results

Results of runs 44 and 45 confirmed the results of runs 13, 18, 19, 22, and 29 (6). The latter series of runs proved that a good filtration was possible with a sub-cooled liquid and without vibration. Runs 44

and 45, which were conducted with sub-cooled liquid, no vibration, and the same cylindrical filter element, indicate that good filtration is possible under these conditions. The carbon dioxide content of the filtrate of runs 44 and 45 was 0.4 ppm, which is lower than the presently known solubility of carbon dioxide of 2.7 ppm at -320°F. In considering this low result, it should be noted that the accuracy of the carbon dioxide measurements with a non-dispersive infrared analyzer is plus or minus 2.0 ppm. (See Table I.)

Runs 46, 47, and 48 were conducted with sub-cooled liquid oxygen with extraneous vibration. The filtrate became cloudy upon tapping and the carbon dioxide content in the filtrate rose correspondingly. These three runs confirm earlier observations as to the effect of vibration of the filter. Figures 3 and 4 illustrate the filter assembly in operation before and after vibration, respectively.

Runs 49 and 50 were conducted with liquid oxygen at its boiling point without extraneous vibration. Vapor and liquid were filtered simultaneously with approximately equal volumes of vapor and liquid in the filter assembly. Approximately 45 ppm of carbon dioxide were found in the filtrate. The vapor and liquid phases emerging from the filter-house were fed into the 1/4-inch O.D. sample coil as with previous runs. The filter-house was partly submerged in the liquid nitrogen, while the coil was completely submerged. The vapor phase was therefore condensed in the coil. From these last runs, it can be concluded that the presence of a vapor phase in the filter-house volume has a considerable effect on the filtration, probably effectively adding to the turbulence effect.

Run 51 was performed to check the possibility of carbon dioxide build-up in the sample coil. The boiling liquid was filtered as in Runs 49 and 50. During filtration the feed to the filter-house was changed to sub-cooled liquid and the filter-house was completely submerged in liquid nitrogen. After filtering under sub-cooled conditions, a sample was trapped in the coil. The analysis showed 37 ppm of carbon dioxide. This was higher than expected, since there was no vibration and the liquid was sub-cooled at the time of sample trapping. This seems to indicate that carbon dioxide is being trapped in the sample coil.

Run 52 was then undertaken to check the carbon dioxide content in the filtrate under sub-cooled conditions without vibration. In this run, the liquid was sub-cooled from the start of the test, and the carbon dioxide analysis, showing 4 ppm carbon dioxide, confirmed the results obtained in Runs 44 and 45. No explanation was found for the high carbon dioxide content of 37 ppm in Run 51. Although it is quite possible that an experimental error may have occurred during the many manipulations associated with each run, no obvious error was apparent in Run 51. This run will be repeated at a later date to confirm this peculiarity.

There are three possible explanations for the occurrence in Run 51:

a. Solid carbon dioxide was accumulating in the coil during the filtration of vapor and liquid. Perhaps the carbon dioxide particles were sticking to the tube wall. These particles were not washed out completely with the filtrate from the sub-cooled filtration.

b. Carbon dioxide did not build up in the coil during filtration of vapor and liquid, but the filtration of sub-cooled liquid following the filtration of vapor and liquid was not as efficient as filtration of sub-cooled liquid from the very start.

c. The combination of (a) and (b) took place. Additional discussion of these phenomena follows in Runs 58, 59, and 60.

Runs 53 through 57 were performed to investigate the influence of turbulence in the filter-house at the cake location. Since a Reynolds number higher than 500 could not be readily obtained in the laboratory-sized filter-house in its original design, the side take-off was added to the filter-house as shown in Figure 2. The main portion of the flow was passed from the inlet to the side take-off without going through the filter element. A small portion of the liquid was passed through the filter element and into the sample coil for analysis. Runs 53, 54, and 55, (Table I), are considered to be of doubtful value since the duration of the tests was about 45 seconds. This time is short, considering the opening and closing of valves, and the reading of liquid levels.

Run 56 was conducted with a 6-1/8 inch O.D. copper feed container instead of the 3-1/8 inch O.D. copper vessel previously used. (See Figure 3) This increased the available feed and therefore duration of the test by a factor of four. The data from this run are termed reliable and the concentration of carbon dioxide in the filtrate fits the correlation fairly well.

Run 57 was performed to duplicate the results of Run 56. However, during the cleaning of the filter element, three large pores were found by using the bubble technique. The three pores were closed with Glyptol and Run 57 gave only 1 ppm carbon dioxide in the filtrate.

The pore size of a porous filter element may be measured by submerging the element in a liquid having a surface tension, σ, for the air-liquid interface and slowly increasing the pressure inside the filter element. Although the air pressure inside the element is slightly higher than the pressure outside the element, no air will pass through the porous metal. Air flow will begin under equilibrium conditions,

$$\Delta P = \sigma/d$$

where ΔP is the minimum differential pressure, σ is the surface tension of the fluid, and d is the inside diameter of the pore. If the pressure inside the element is further increased, a bubble will appear on the filter element. The pressure drop at the first bubble is an indication of the pore size according to the above equation. If the pressure is further increased, bubbles appear on the entire surface of the filter. This is the boiling point of the bubble test (9). The filter element in question was bubble-tested qualitatively after Run 56. Before the boiling point was reached, three holes were bubbling. These holes were patched before starting Run 57.

The size of these holes was so different from the average pore size that the air flowing out could be distinctly felt with the finger tip. No attempt was made to measure the pressure drop for these holes but from earlier experience with bubble tests, the holes were estimated to be larger than 100 microns in diameter.

It must be realized that all the previous runs discussed in this report were done with the holes present. Since the question of whether the holes had any effect on the filtration of boiling liquid arises immediately, Run 58 was performed with the same holes still plugged at conditions similar to Runs 49 and 50. The carbon dioxide content in the filtrate was 88 ppm, indicating that the three large holes had no effect on the filtration of the boiling liquid. The filtrate in this test contained twice the amount of carbon dioxide as in Runs 49 and 50. The boiling conditions cannot be duplicated, especially since the ratio of vapor to liquid can only be kept constant within certain limits. Runs 59 and 60 were performed to further investigate the cause of the build-up of carbon dioxide in the coil during a "boiling" run. These runs were made to check any possible relationship between the amount of carbon dioxide build-up in the coil and the amount of filtered liquid and vapor.

B. Hydrocarbon Film Ignition Work

Due to the urgency of preparing for and studying the sampling and analysis program for liquid oxygen which is being conducted at Cape Canaveral by members of the Research and Development Staff of Air Products and Chemicals, Inc., no work was performed on hydrocarbon film ignition during this period. It is expected that, as of approximately March 1, this work will be resumed with emphasis being placed on obtaining an allowable film concentration based on an adequate statistical analysis of these tests.

C. Filter Investigation

There has been considerable discussion in the past as to the efficacy of the various types of filtering media and the factors affecting their application in the filtration of particulate matter from cryogenic fluids. As part of the subject contract, Air Products and Chemicals has reviewed the filter market and herein makes a recommendation of the type of filter which should be used in the missile program, particularly in the filtration of solids from liquid oxygen.

Filters suitable for this purpose fall in three general classes: woven wire mesh, sintered metal powders rolled into sheet form, and porous ceramic filters.

Each of these types is now available in all size ranges from approximately 1-micron nominal pore size through the range of 175-micron nominal size. The quality control on these filters is adequate in each case and for normal ambient temperature operations, the three may be considered essentially interchangeable, provided no weight criterion is involved. If there is a weight limitation, the use of the porous ceramic filter may not be suitable, since this filter is by design necessity a thicker, heavier unit. As the filtering temperature departs farther from ambient temperature, a thermal shock is applied to the filter medium at the time that the first amount of cryogenic fluid contacts the filter surface. This impact of cryogenic fluid causes a sudden contraction of the material in locations where the cryogenic fluid first contacts the filter unit. When a filter unit of the mass thich is found in the sintered ceramic or alumina filter is hit by a cryogenic liquid, spalling of small areas of the surface may occur. If the spalling should occur because of sudden compressive stresses on the inside of the filter or on the downstream side of the filter, additional particles detrimental to the operation of the missile liquid oxygen system could be liberated and carried into the system. These pieces would be doubtless far greater than any of the now known limitations which have been applied to particulate matter and could cause serious trouble if allowed to penetrate into the missile system.

In contrast to this situation, the woven wire cloth, which is much more integral or homogeneous, will not provide this spalling tendency. The material of this filter medium is therefore acceptable to the process and procedures involved in the transfer of missile liquid oxygen. It should be noted that in the liquid oxygen generating units in operation in this country, both porous ceramic filters and sintered metal filters are in use. However, during the operation of these filter assemblies in the oxygen generating plant, sufficient time is allowed for slow cooling of the filter elements by the slow introduction of cold material. In these situations, the use of the sintered material is satisfactory. However, in the transfer of missile liquid oxygen, there is no possible allowance for a cooling period. Therefore, the full thermal shock of liquid oxygen at -297°F contacting the warm (70°F) filter element must be considered.

The previous progress report of October, 1960 (6) and this report show that the filtration of carbon dioxide cannot be adequately performed under the flow and temperature conditions found in missile liquid oxygen transfer systems. Since such is the case, no attempt has been made to determine the design which will fill the requirements set forth in the contract. During the performance of the contract, Air Products and Chemicals was not able to obtain commitments from the missile and rocket engine manufacturers and missile sub-system manufacturers as to the maximum allowable size of the particulate matter for their systems. It is understood by Air Products and Chemicals that this situation also exists within the framework of the Air Force Systems Command and that steps are being taken and will be taken to experimentally check various missile systems with cryogenic fluids containing larger particulate matter in an attempt to ease the stringent requirements now in force.

The Appendix lists the manufacturers of filter media which were used in the solid carbon dioxide filtering tests. These represent a cross-section of the manufacturers of the various types of filter media which were considered possibly satisfactory for filtering cryogenic liquids. This is not a complete list of all manufacturers of such equipment in the United States.

III. CONCLUSIONS

The solid carbon dioxide filtration tests which were performed during this report period justify our previous position that solid carbon dioxide cannot be successfully filtered out of liquid oxygen under the flow conditions which exist at the time of transfer at a missile site. Boiling liquid is handled at transfer rates which cause a great deal of turbulence; thus, solid carbon dioxide will be transmitted to the filter and into the container. In order to perform such a filtration, it would be necessary to supply a quiescent pool of liquid in which to install the filter and a very slow filtration rate to enable the solid carbon dioxide filter cake to build up on the surface of the filter.

Data at the higher Reynolds numbers as shown in Fig. 1 represent conditions found in the actual generating plant and provide additional strength for the proposal of the carbon dioxide concentration versus Reynolds number correlation.

IV. FUTURE WORK

A. Carbon Dioxide

It is planned to continue the carbon dioxide filtration tests under more closely controlled conditions to attempt to narrow the spread of data. These tests will be performed again in the full span of laboratory conditions, primarily under quiescent conditions in handling the sub-cooled contaminated oxygen liquid.

B. Hydrocarbon Film Ignition

The work will be resumed about March 1, 1960 with emphasis placed on obtaining an allowable film concentration based on adequate statistical analysis of these tests.

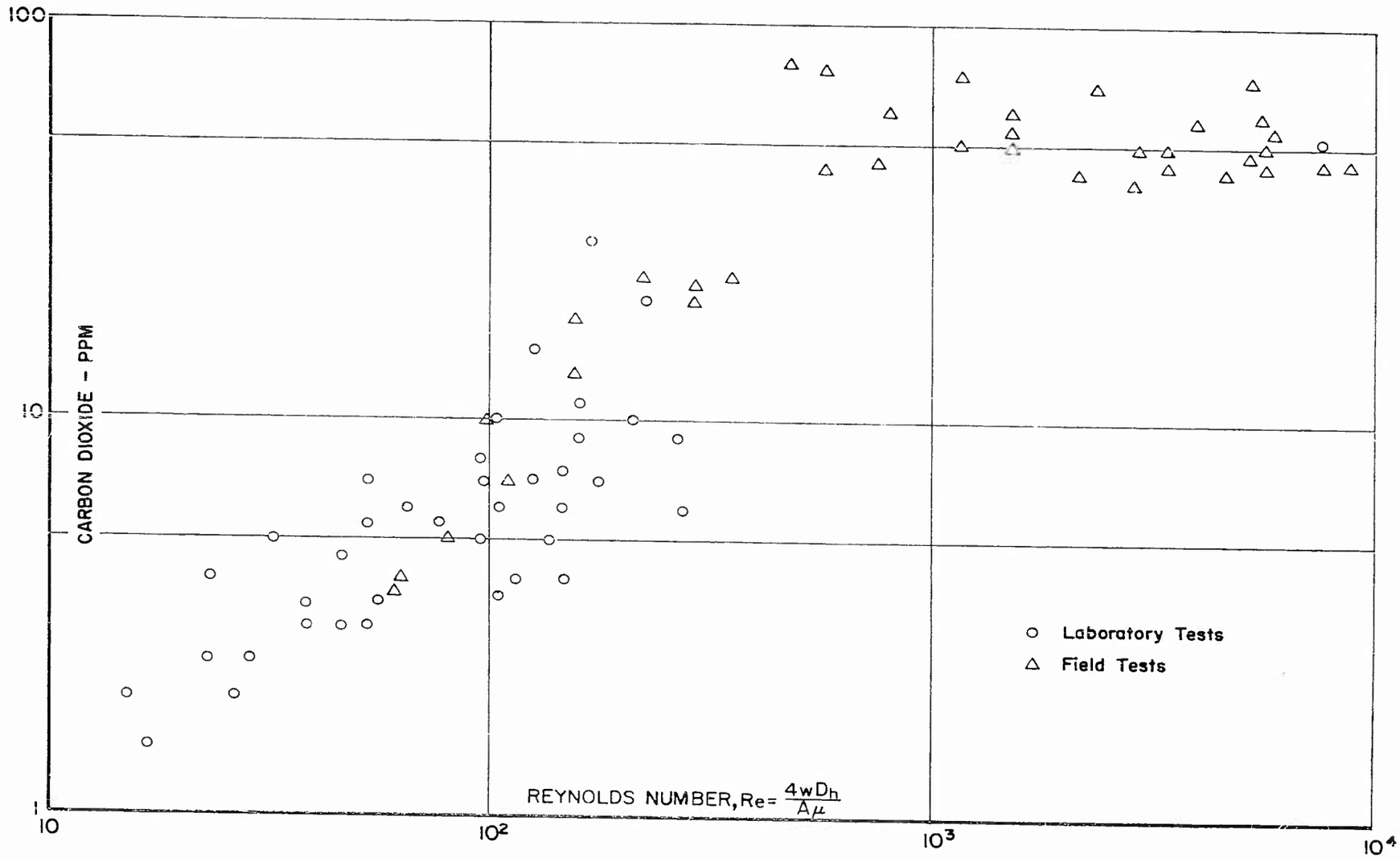

Figure 1. The Variation of Carbon Dioxide After Filtration to Reynolds Number in Filter Assembly.

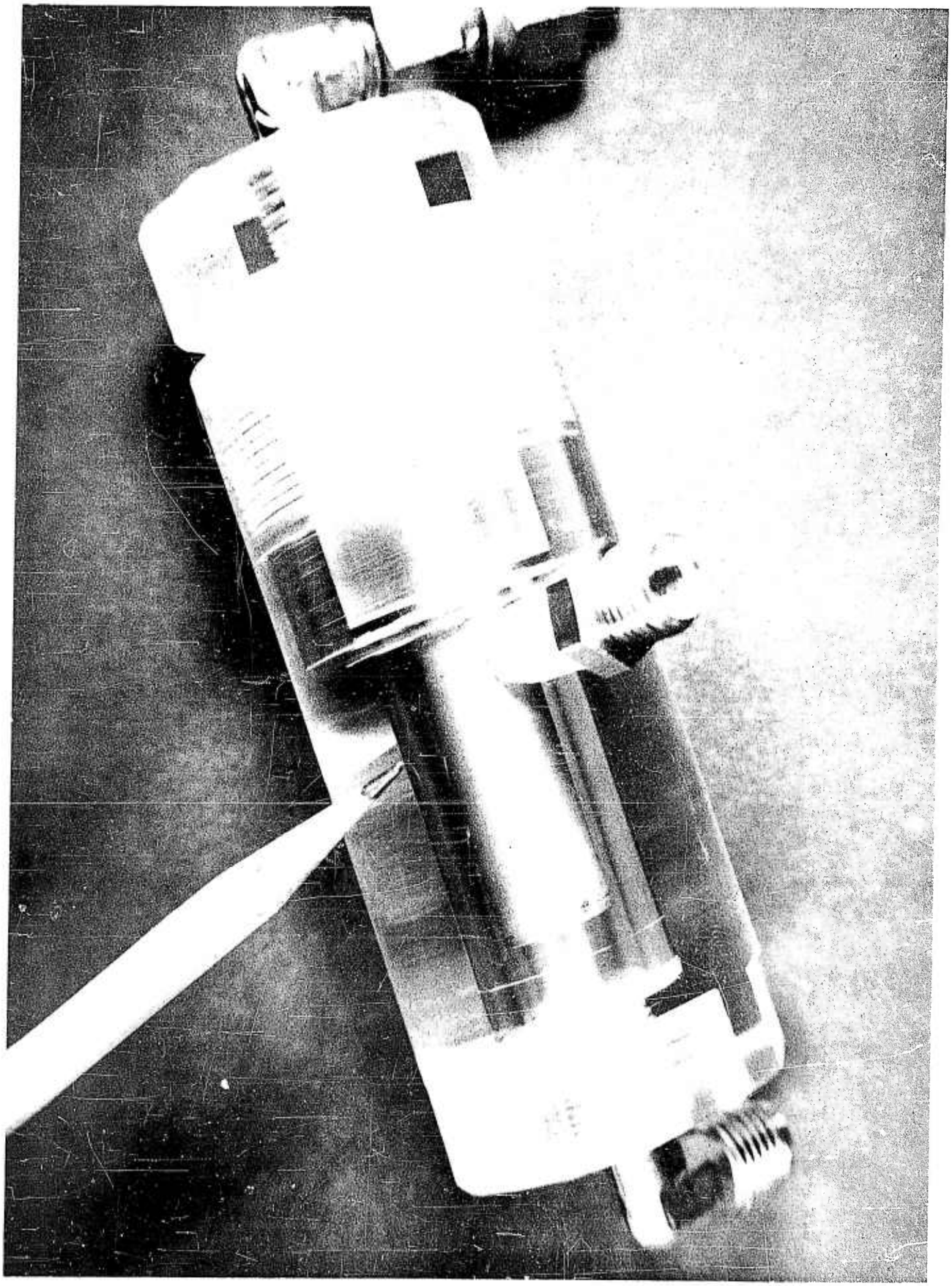

FIGURE 2. NEW FILTER ASSEMBLY WITH SIDE TAP

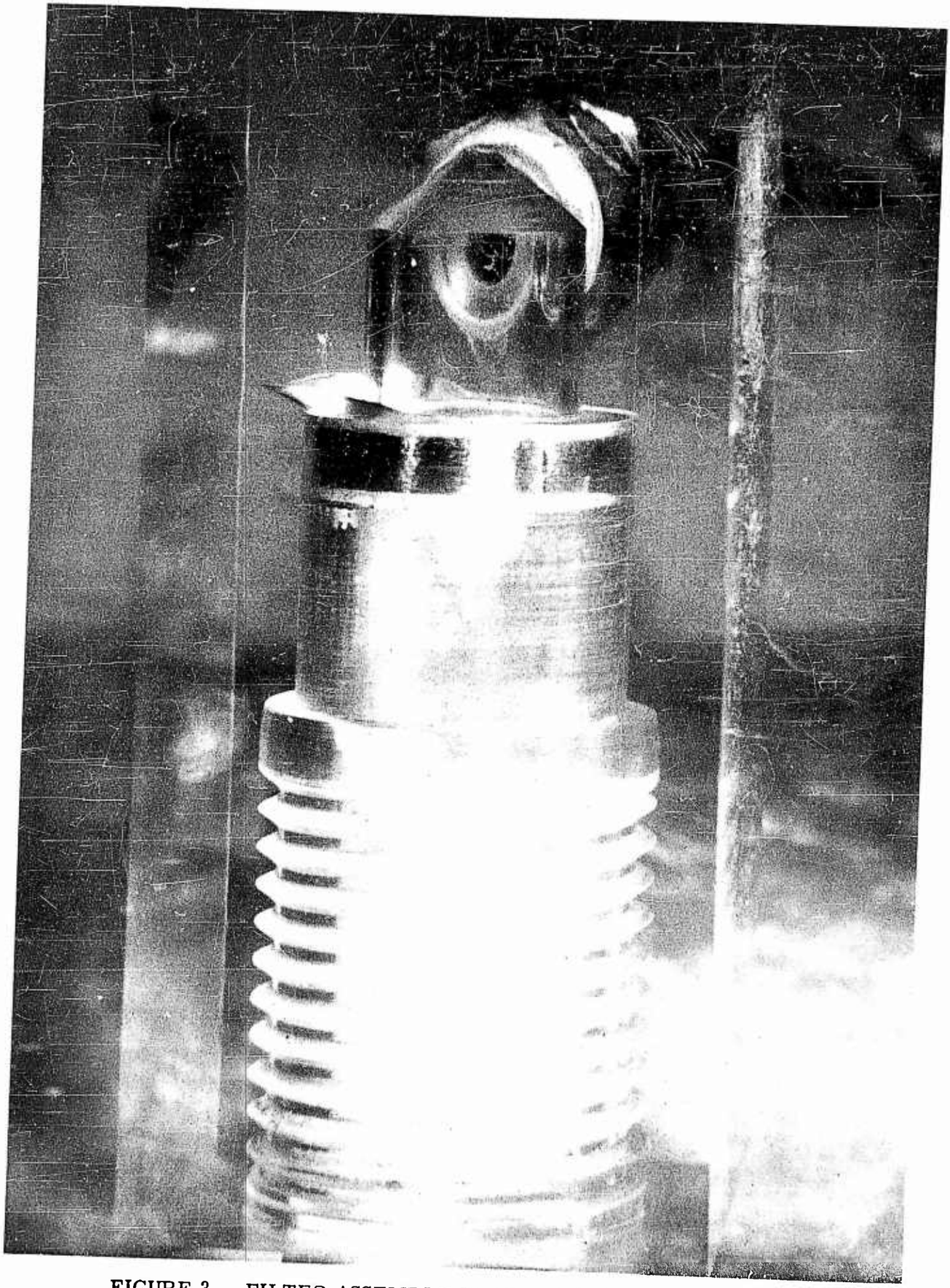

FIGURE 3. FILTER ASSEMBLY IN OPERATION - BEFORE VIBRATION

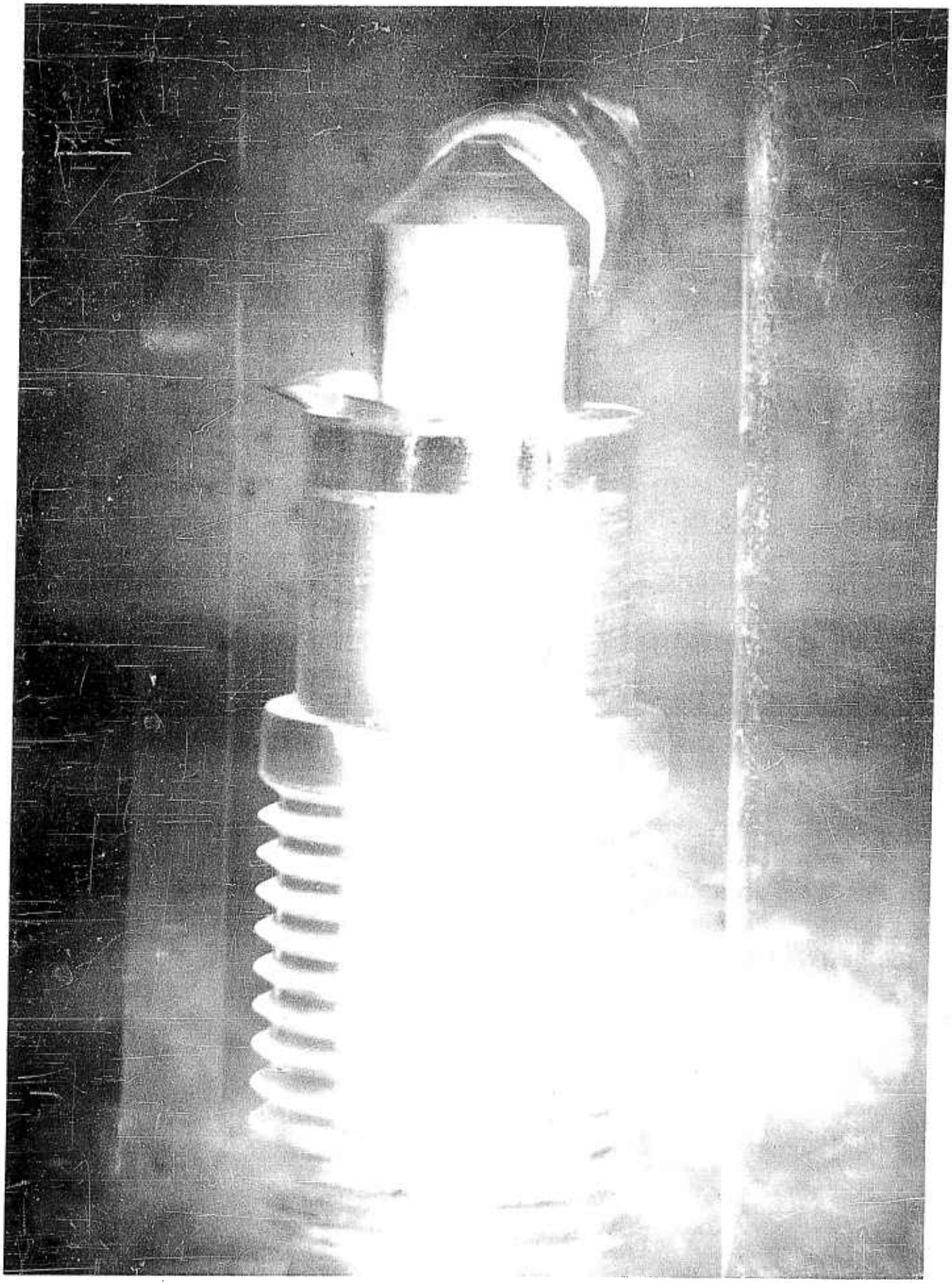

FIGURE 4. FILTER ASSEMBLY IN OPERATION - DURING VIBRATION

TABLE I.

SUMMARY OF FILTER TESTS DATA*

Porous Stainless Steel Filter

Run No.	△P psi	Reynolds Number	Liquid Velocity ft/hr	CO_2 ppm	Pore Size microns	Remarks
44	4	75	2.9	0.4	5	Subcooled, no vibration.
45	4	147	5.7	0.4	7	Subcooled, no vibration.
46	4	71	2.7	78	7	Subcooled, vibration. Filtrate cloudy during tapping.
47	4	41	1.6	38	5	Subcooled, vibration. Filtrate cloudy during tapping.
48	4	67	2.6	106	5	Subcooled, vibration. Filtrate cloudy during tapping.
49	4	---	---	45	5	Boiling. Equal volume vapor/liquid in filter house.
50	4	---	---	46	5	Boiling.
51	4	43	1.6	37	5	Boiling at start. Sample taken subcooled.
52	4	23	.9	0.4	5	Subcooled, no vibration.
53	10	4450	---	---	5	Run failed. High Re by side take-off.
54	40	3250	---	(1.5)	5	Run uncertain.
55	40	4450	11.0	(36)	5	Run failed. High Re by side take-off.
56	-	13500	10.3	40	5	6 1/8" O.D. Feed Container. High Re number.
57	-	12100	---	1	5	Patched up three holes in filter element.
58	4	---	---	88	5	Boiling.
59	4	---	---	19	5	Boiling.
60	4	---	---	23	5	Boiling.

Not reliable runs.

* Upstream CO_2 Concentration - 1000 ppm.

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APPENDIX

LIST OF FILTER VENDORS

Aircraft Porous Media, Incorporated
Glen Cove, New York

Arno Engineering Corporation
Meriden, Connecticut

Bendix Filter Division, Bendix Aviation Corporation
Madison Heights, Michigan

Carborundum Company
Perth Amboy, New Jersey

Commercial Filters Corporation
Melrose, Massachusetts

Dollinger Corporation
Rochester, New York

Harmon Equipment Company
Los Angeles, California

Micro Metallic Division, Pall Corporation
Glen Cove, New York

Purolator Products, Incorporated
Rahway, New Jersey

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Development Center	58
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Callery Chemical Company	65
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Fulton-Irgon Corporation	70

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Hughes Tool Company	73
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Liquid Propellant Information Agency	75 to 77
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Monsanto Chemical Company	79
New York University	80
Olin Mathieson Chemical Corporation, New Haven	81 to 83
Peninsular ChemResearch Corporation	84
Pennsalt Chemicals Corporation	85
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